

Standard Operating Procedure (SOP) Interim Change Notice (ICN)

Effective Date: 4/30/04

20 Pages

Section 1: Description of Change (Requester completes)

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2. SOP 15.05, R1

3. ICN No.: 1

4. SOP Title: Routine Validation of Inorganic Data

5. Description of Change:

Updated IF/THEN tables to match checklist lines. (See attached.)

6. Attachments Modified, Added, or Removed:

☐ Yes

☒ No

7. ICN Justification:

Table reference to checklist was off by one line.

8. Requester: John Wilcox

[\[Signature on File\]](#)

(Print name, then sign.)

4/21/04

(Date)

Section 2: Evaluation and Approval (PTL, Technical Reviewer, and QPPL complete)

9. Evaluation Remarks: (If none, enter N/A)

NA

10. Team Leader: Stephen Bolivar

[\[Signature on File\]](#)

(Print name, then sign.)

4/21/04

(Date)

11. Technical Reviewer: Felicia Aguilar

[\[Signature on File\]](#)

(Print name, then sign.)

4/21/04

(Date)

12. QPPL: Phil Noll

[\[Signature on File\]](#)

(Print name, then sign.)

4/21/04

(Date)

QP-4.1, R5

Los Alamos National Laboratory
RRES-Remediation Services

[Using a token card, click here to record "self-study" training to this procedure.](#)

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

6.3 Verifying Calibrations

Verify the presence of the initial- and continuing -calibration verification (ICV and CCV) results using the forms supplied by the analytical laboratory.

1. IF ICV and CCV analysis documentation for each sample matrix and/or analytical batch is...	THEN...
Present,	<ul style="list-style-type: none">Record "No" on line 4-3 of the Inorganic Data-Validation Checklist.Go to Step 2.
Missing,	<ul style="list-style-type: none">Record "Yes" on line 4-3 of the Inorganic Data-Validation ChecklistContact the laboratory and SMO to request the missing information (see Section 6.1-4).If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I16) on the individual sample Form 1.Go to Step 2.

Table 6.3-1
Calibration Verification Limits

Analytes	LAL	LWL	UWL	UAL
All (except Hg and CN)	75	90	110	125
Hg	65	80	120	135
CN	70	85	115	130
LAL—Lower Acceptable Limit, LWL—Lower Warning Limit, UWL—Upper Warning Limit, UAL—Upper Acceptable Limit, Hg—mercury, CN—cyanide				

2.	IF...	THEN...
	<u>All</u> the ICV or CCV percent recoveries (%Rs) are = the upper acceptably limit (UAL) listed in table 6.3-1 ,	<ul style="list-style-type: none"> Record “No” on line 5-4 of the Inorganic Data-Validation Checklist. Go to Step 3.
	<u>Any</u> ICV or CCV %R are > the UAL,	<ul style="list-style-type: none"> Record “Yes” on line 5-4 of the Inorganic Data-Validation Checklist and for the affected analytes. Qualify detected analytes as rejected (R, I16a) on the individual sample Form 1. Go to Step 3.

3.	IF...	THEN...
	<u>No</u> ICV %R is > the upper warning limit (UWL),	<ul style="list-style-type: none"> Record “No” on line 6-5 of the Inorganic Data-Validation Checklist. Go to Step 4.
	<u>Any</u> ICV or CCV %R is > UWL and = the UAL,	<ul style="list-style-type: none"> Record “Yes” on line 6-5 of the Inorganic Data-Validation Checklist. For the affected analytes qualify the result for each detected analyte as estimated with a potential positive bias (J+, I16b) in the associated samples on the individual sample Form 1. Go to Step 4.

4.	IF...	THEN...
	<u>No</u> ICV or CCV %R is < the lower warning limit (LWL),	<ul style="list-style-type: none"> Record "No" on line 7-6 of the Inorganic Data-Validation Checklist. Go to Step 5.
	<u>Any</u> ICV or CCV %R is < the LWL and = the lower acceptable limit (LAL),	<ul style="list-style-type: none"> Record "Yes" on line 7-6 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each detected analyte as estimated with a potential negative bias (J-, I16c) in the associated samples on the individual sample Form 1. For the affected analytes, qualify the results for each undetected analyte as estimated (UJ, I16c) in the associated samples on the individual sample Form 1. Go to Step 5.

5.	IF...	THEN...
	<u>No</u> ICV or CCV %R is < the LAL,	<ul style="list-style-type: none"> Record "No" on line 8-7 of the Inorganic Data-Validation Checklist. Go to Step 6.
	<u>Any</u> ICV or CCV %R is < the LAL,	<ul style="list-style-type: none"> Record "Yes" on line 8-7 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each analyte as rejected (R, I16d) in the associated samples. Go to Step 6.

6. IF...	THEN...
The calibration correlation coefficient is < 0.995 for multipoint calibration analytes (ICPMS, CN, wet chem.),	<ul style="list-style-type: none"> Record "Yes" on line 9-8 of the Validation Checklist. Qualify the results for all analytes as rejected (R, I16e) in the associated samples on the individual sample Form 1. Go to Section 6.4, "Verifying ICPES Interference Check Sample Results."
Multipoint calibrations are <u>not</u> used <u>or</u> if the calibration coefficient is acceptable,	<ul style="list-style-type: none"> Record "No" on line 9-8 of the Inorganic Data-Validation Checklist. Go to Section 6.4, "Verifying ICPES Interference Check Sample Results."

6.4 Verifying ICPES Interference Check Sample Results

Verify the presence of the ICS %R values using forms provided by the analytical laboratory. The ICS must contain the following analytes: Ag, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb (see note, below), V and Zn. The QC acceptance limits are $\pm 20\%$.

Note: If lead was analyzed by graphite furnace atomic absorption (GFAA), no ICS result is required. This information should be noted in the comment section of the Data-Validation Cover Sheet.

1.	IF the ICS documentation is...	THEN...
	<u>Present</u> for each sample matrix and/or analytical batch,	<ul style="list-style-type: none"> Record "No" on line 10-9 of the Inorganic Data-Validation Checklist. Go to Step 2.
	<u>Missing</u> for each sample matrix and/or analytical batch,	<ul style="list-style-type: none"> Record "Yes" on line 10-9 of the inorganic data-validation checklist. Contact the laboratory and the SMO to request the missing information (see Section 6.1 -4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I7) on the individual sample Form 1. Go to Step 2.

2.	IF...	THEN...
	<u>All</u> ICS %R is = 120%,	<ul style="list-style-type: none"> Record "No" on line 11-10 of the Inorganic Data-Validation Checklist. Go to Step 3.
	<u>Any</u> ICS analyte %R value is > 120%	<ul style="list-style-type: none"> Record "Yes" on line 11-10 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each detected analyte as estimated with a potential positive bias (J+, I7a) in the associated samples. Go to Step 3.

3.	IF...	THEN...
	<u>All</u> ICS %R is = 80%,	<ul style="list-style-type: none"> Record "No" on line 12-11 of the Inorganic Data-Validation Checklist. Go to Step 4.

3. IF...	THEN...
<u>Any</u> ICS %R is = 50% and < 80%,	<ul style="list-style-type: none"> Record "Yes" on line 12-11 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each detected analyte, as estimated with a potential negative bias (J-, I7b) in the associated samples on the individual sample Form 1, <p style="text-align: center;">OR</p> <p>For the affected analytes qualify the results for each undetected analyte as estimated (UJ, I7b) in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 4.

4. IF...	THEN...
<u>All</u> ICS %R values are = 50%,	<ul style="list-style-type: none"> Record "No" on line 13-12 of the Inorganic Data-Validation Checklist. Go to Section 6.5, "Verifying the Matrix Spike Results."
<u>Any</u> ICS analyte %R value is < 50%,	<ul style="list-style-type: none"> Record "Yes" on line 13-12 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results as rejected (R, I7c) for each analyte in the associated samples on the individual sample Form 1 Go to Section 6.5, "Verifying the Matrix Spike Results."

6.5 Verifying the Matrix Spike Results

Verify the presence of the matrix spike (MS) sample %R values using the forms provided by the analytical laboratory. The MS acceptance criteria are 75%–125%, inclusive for all spiked analytes.

Note: If the sample result is greater than four times the spike added for any analyte, these acceptance criteria do not apply (per the applicable methods) for that analyte.

1.	IF an MS was...	THEN...
	Analyzed on a sample associated with this request and the MS included all required analytes,	<ul style="list-style-type: none"> Record "No" on lines 4413, 4514, and 15 of the Inorganic Data-Validation Checklist. Go to Step 4.
	<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 4413 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I3) on the individual sample Form 1. Go to Step 2.

2.	IF...	THEN...
	If insufficient sample volume was submitted for analysis and no MS could be analyzed,	<ul style="list-style-type: none"> Record "Yes" on line 4514 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I3a) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3a) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 3.
	If the sample volume is sufficient,	<ul style="list-style-type: none"> Go to Step 3.

3.	IF the MS was performed on a...	THEN...
	Non-LANL sample,	<ul style="list-style-type: none"> Record "Yes" on line 16 15 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I3b) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3b) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 4.
	LANL sample,	<ul style="list-style-type: none"> Go to Step 4.

4.	IF...	THEN...
	All MS %R are = 150%,	<ul style="list-style-type: none"> Record "No" on line 17 16 of the Inorganic Data-Validation Checklist. Go to Step 5.
	Any MS %R is > 150%,	<ul style="list-style-type: none"> Record "Yes" on line 17 16 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated with a potential positive bias (J+, I3c) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3c) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 5.

5.	IF...	THEN...
	<u>All</u> MS %R are = 125%,	<ul style="list-style-type: none"> Record "No" on line 18-17 of the Inorganic Data-Validation Checklist. Go to Step 6.
	<u>Any</u> MS %R is > 125% and = 150%,	<ul style="list-style-type: none"> Record "Yes" on line 18-17 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated with a potential positive bias (J+, I3d) for each detected analyte in the associated samples on the individual sample Form 1. Go to Step 6.

6.	IF...	THEN...
	<u>All</u> MS %R = 75%,	<ul style="list-style-type: none"> Record "No" on line 19-18 of the Inorganic Data-Validation Checklist. Go to Section 6.6, "Verifying Duplicate Sample Analysis Results."
	<u>Any</u> MS %R is = 30% and is < 75%,	<ul style="list-style-type: none"> Record "Yes" on line 19-18 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated potential negative bias (J-, I3e) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3e) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 7.

7. IF...	THEN...
<u>All</u> MS %R are = 30%,	<ul style="list-style-type: none"> Record "No" on line 20-19 of the Inorganic Data-Validation Checklist. Go to Section 6.6, "Verifying Duplicate Sample Analysis Results."
<u>Any</u> MS %R is < 30%,	<ul style="list-style-type: none"> Record "Yes" on line 20-19 of the Inorganic Data-Validation Checklist. Qualify the affected results as rejected (R, I3f) for each analyte in the associated samples on the individual sample Form 1. Go to Section 6.6, "Verifying Duplicate Sample Analysis Results."

6.6 Verifying Duplicate-Sample Analysis Results

Verify the presence of the analytical laboratory duplicate-sample %R values using the forms provided by the analytical laboratory. If the sample and duplicate-sample results are greater than or equal to five times the RL, the duplicate-sample criterion for aqueous samples is an RPD less than or equal to 20%; the duplicate-sample criterion for solid samples is an RPD less than or equal to 35%. If either the sample or duplicate-sample value is less than five times the RL, a control limit must be used that is equal to the RL for water samples and two times the RL for solid samples.

1. IF a duplicate sample was...	THEN...
Analyzed on a sample associated with this request and the duplicate-sample analysis included all required analytes,	<ul style="list-style-type: none"> Record "No" on lines 2420, 2221, and 22 of the Inorganic Data-Validation Checklist. Go to Step 4.
<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 24-20 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information

	<p>(see Section 6.1-4).</p> <ul style="list-style-type: none"> • If the laboratory cannot provide the missing information, qualify the affected results as estimated for duplicates and serial dilutions are not as critical to overall data usability. (J, I10/ UJ, I10) on the individual sample Form 1. • Go to Step 2.
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2.	IF...	THEN...
	An insufficient sample volume was submitted for analysis and no duplicate sample could be analyzed,	<ul style="list-style-type: none"> • Record "Yes" on line 22-21 of the Inorganic Data-Validation Checklist. • Qualify the affected results as estimated (J, I10a/UJ, I10a) on the individual sample Form 1. • Go to Step 3.

3.	IF the duplicate was performed on a...	THEN...
	Non-LANL sample,	<ul style="list-style-type: none"> • Record "Yes" on line 23-22 of the Inorganic Data-Validation Checklist. • Qualify the affected results as estimated (J, I10b/UJ, I10b) on the individual sample Form 1. • Go to Step 4.
	LANL sample,	<ul style="list-style-type: none"> • Go to Step 4.

4.	IF the...	THEN...
	Duplicate sample meets all QC criteria,	<ul style="list-style-type: none"> • Record "No" on lines 24-23 and 25-24. • Go to Section 6.7, "Verify Laboratory Control Sample"

	Results.”
Sample result and the duplicate sample result are each = five times the RL, <u>and</u> the RPD exceeds 20% for aqueous samples <u>or</u> 35% for soil samples,	<ul style="list-style-type: none"> Record “Yes” on line 24-23 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I10c/UJ, I10c) on the individual sample Form 1. Go to Step 5.

5. If either the sample result or the duplicate sample result is less than 5 times the RL, and the difference between the sample result and the duplicate sample result is greater than the RL for water samples or greater than 2 times the RL for solid samples,

- A. Record “Yes” on line 25 of the Inorganic Data-Validation Checklist.
- B. Qualify the affected results as estimated (J, I10d/UJ, I10d) on the individual sample Form 1.
- C. Go to Section 6.7, “Verifying Laboratory Control Sample Results.”

6.7 Verifying Laboratory Control Sample Results

Verify the presence of the laboratory control sample (LCS) %R values using forms provided by the analytical laboratory. The LCS criteria to apply to soil and water samples are given in Table. 6.7-1.

Table 6.7-1
Laboratory Control Sample Recovery Criteria

Analyte	LAL	LWL	UWL
Soil	30	75	125
Water	50	80	120
All values in % recovery (%R)			

Note: The solid LCS recovery criteria do not apply to Ag or Sb.

1.	IF an appropriate LCS was...	THEN...
	Analyzed and reported,	<ul style="list-style-type: none"> Record "No" on line 26-25 of the Inorganic data-validation checklist. Go to Step 2.
	<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 26-25 of the inorganic data-validation checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I6) on the individual sample Form 1. Go to Step 2.

2.	IF...	THEN...
	<u>All</u> LCS %R are = the LWL,	<ul style="list-style-type: none"> Record "No" on line 27-26 of the Inorganic Data-Validation Checklist. Go to Step 3.
	<u>Any</u> LCS %R values are > the UWL,	<ul style="list-style-type: none"> Record "Yes" on line 27-26 of the Inorganic Data-Validation Checklist. Qualify all detected results associated with the high LCS recovery in the affected samples as estimated with a potential high bias (J+, I6a) on the individual sample Form 1. Go to Step 3.

3. IF...	THEN...
<u>All</u> LCS %R are = the LWL,	<ul style="list-style-type: none"> Record "No" on line 28-27 of the Inorganic Data-Validation Checklist. Go to Step 4.
<u>Any</u> LCS analyte %R value is = the LAL and < the LWL,	<ul style="list-style-type: none"> Record "Yes" on line 28-27 of the inorganic data-validation checklist. For each detected analyte qualify the affected results as estimated with a potential negative bias (J-, I6b) on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>For each undetected analyte qualify the affected results as estimated with a potential low bias (UJ, I6b) on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 4.

4. IF...	THEN...
<u>All</u> LCS %R are = the LWL,	<ul style="list-style-type: none"> Record "No" on line 29-28 of the Inorganic Data-Validation Checklist. Go to Section 6.8, "Verifying Serial Dilution Sample Results."
<u>Any</u> LCS analyte %R value is < the LAL,	<ul style="list-style-type: none"> Record "Yes" on line 29-28 of the Inorganic Data-Validation Checklist. Qualify the affected results as rejected (R, I6c) on the individual sample Form 1. Go to Section 6.8, "Verifying Serial Dilution Sample Results."

6.8 Verifying Serial Dilution Sample Results

Verify the presence of the serial dilution sample %R values using forms provided by the analytical laboratory. A serial dilution must be analyzed for inductively coupled plasma atomic emission spectroscopy (ICPAES) and

for inductively coupled plasma mass spectroscopy (ICPMS). The serial dilution must be performed on a sample from each group of samples with a similar matrix type (for example, water, soil).

Note: Samples identified as field blanks cannot be used for the serial dilution sample.

Note: If the analyte concentration is sufficiently high (greater than 50 times the IDL for ICPAES and greater than 100 times the IDL for ICPMS), the serial dilution sample (a fivefold dilution) must then agree within a 10% relative difference of the original sample value after correction for the dilution.

Note: The qualifier for a missing serial dilution sample is J, estimated. This appears inconsistent with qualifying data with other missing QC documentation as R, rejected. The difference lies in the fact that the R qualifiers are assigned due to shortfalls in calibration requirements. The end user of the data should determine whether a greater impact is seen on sample results when a serial dilution sample is missing.

1. IF a serial dilution sample was...	THEN...
Analyzed and reported,	<ul style="list-style-type: none"> Record "No" on lines 30-29 and 34-30 of the Inorganic Data-Validation Checklist. Go to Step 3.
<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 30-29 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1 -4). If the laboratory cannot provide the missing information, qualify the affected results as estimated (J, I18/UJ, I18) on the individual sample Form 1. Go to Step 2.

2.	IF a serial dilution sample was performed on a...	THEN...
	Non-LANL sample,	<ul style="list-style-type: none"> Record "Yes" on line 34-30 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I18a/UJ, I18a) on the sample Form 1. Go to Step 3.
	LANL sample,	<ul style="list-style-type: none"> Go to Step 3.

3.	IF...	THEN...
	<u>All</u> serial dilution %R are = 10%,	<ul style="list-style-type: none"> Record "No" on line 32-31 of the Inorganic Data-Validation Checklist. Go to Section 6.9, "Verifying Holding Times."
	<u>Any</u> serial dilution sample %R values are > 10% and the analyte value is > 50 times the MDL in the original sample for ICPAES analytes (> 100 times the MDL for ICPMS),	<ul style="list-style-type: none"> Record "Yes" on line 32-31 of the Inorganic Data-Validation Checklist. Qualify the results as estimated (J, I18b/UJ, I18b) on the individual sample Form 1. Go to Section 6.9, "Verifying Holding Times."

6.9 Verifying Holding Times

Note: Holding times for metals (except cyanide and mercury in aqueous samples) are typically six months. The holding time for mercury in an aqueous sample is 28 days after the sample collection. The holding time for cyanide in an aqueous sample is 14 days after the sample collection. Applicable storage conditions are found in the current SOW for analytical services (LANL 1995b).

1.	IF...	THEN...
	<u>All</u> samples were analyzed within the prescribed holding time,	<ul style="list-style-type: none"> Record "No" on line 33-32 of the Inorganic Data-Validation Checklist. Go to Section 6.10, "Identifying

	the Detection Status.”
<u>Any</u> of the samples were <u>not</u> analyzed within the prescribed holding time,	<ul style="list-style-type: none"> Record “Yes” on line 33-32 of the Inorganic Data-Validation Checklist. For each detected analyte in the associated samples qualify the results as estimated with a potential negative bias (J-, I9) on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>For each undetected analyte in the associated samples, qualify the results as estimated (UJ, I9) on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 2.

2. IF...	THEN...
<u>All</u> samples were analyzed fewer than two times the prescribed holding time,	<ul style="list-style-type: none"> Record “No” on line 34-33 of the Inorganic Data-Validation Checklist. Go to Section 6.10, “Identifying the Detection Status.”
<u>Any</u> sample was analyzed more than two times the prescribed holding time,	<ul style="list-style-type: none"> Record “Yes” on line 34-33 of the Inorganic Data-Validation Checklist. For each detected analyte in the associated samples, qualify the results as estimated with a potential negative bias (J-, I9a) on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>For each undetected analyte in the associated samples, qualify the results as rejected (R, I9a) on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Section 6.10, “Identifying the Detection Status.”

6.10 Identifying the Detection Status

Note: In order to meet the regulatory requirements imposed upon the RRES-R Program with the technology routinely available from the environmental laboratory community, the RRES-R Program requires analytical laboratories to report inorganic analytes as detected down to the MDL. In order to identify results below the RL and results with greater errors in quantitation, the laboratories have been instructed to apply a “B” flag to all results between the MDL and RL.

IF...	THEN...
<u>No</u> results are reported with a “B” flag,	<ul style="list-style-type: none">Record “No” on line 35-34 of the Inorganic Data-Validation Checklist.Go to Section 6.11, “Identifying Obvious Quality Deficiencies.”
<u>Any</u> results are reported by the contract laboratory with a “B” flag,	<ul style="list-style-type: none">Record “Yes” on line 35-34 of the inorganic data-validation checklist.For each detected analyte qualify affected results as estimated (J, I1) on the individual sample Form 1.Go to Section 6.11, “Identifying Obvious Quality Deficiencies.”

Note: A J,I1 qualifier is superseded by U,I4a.

6.11 Identifying Obvious Quality Deficiencies

IF...	THEN...
<u>Any</u> significant or obvious data-quality deficiencies during the data-validation process are noticed,	<ul style="list-style-type: none">Record “Yes” on line 36-35 of the Inorganic Data-Validation Checklist.Contact the analytical laboratory and SMO, if necessary to resolve the quality issue.Record the appropriate data qualifier based on the validator’s best professional judgment and apply reason code I19.Describe clearly the quality issue flagged on the Data-Validation Cover Sheet.

IF...	THEN...
	<ul style="list-style-type: none"> Go to Section 6.12, "Assembling the Data Record Package."
There are <u>no</u> obvious quality deficiencies outside of those covered by this SOP,	<ul style="list-style-type: none"> Record "No" on line 3635 of the Inorganic Data-Validation Checklist. Go to Section 6.12, "Assembling the Data Record Package."

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Author: John A. Wilcox		

Risk Reduction and Environmental Stewardship— Remediation Program

Standard Operating Procedure

for Routine Validation of Inorganic Data



Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the University of California for the United States Department of Energy under contract W-7405-ENG-36.

Revision Log

<i>Revision No.</i>	<i>Effective Date</i>	<i>Prepared By</i>	<i>Description of Changes</i>	<i>Affected Pages</i>
R0	06/14/00	Bart J. Vanden Plas	Initial Procedure	All
R1	05/21/03	John A. Wilcox	Rewritten to streamline and update process	All

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Routine Validation of Inorganic Data

1.0 PURPOSE

- 1.1 This standard operating procedure (SOP) represents the minimum standards for evaluating routine inorganic analytical data. These data can be generated for the Los Alamos National Laboratory (LANL), Risk Reduction and Environmental Stewardship—Remediation (RRES-R) Program, using U.S. Environmental Protection Agency (EPA) SW-846 Methods 6010, 6020, 7000, 9000, 300 series, or the comparable Contract Laboratory Program (CLP) methods under the current statement of work (SOW) for analytical services. The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used to develop more focused data-validation requirements specific to a particular data use.
- 1.2 Implementation of this procedure results in a tabulation of data compliances and noncompliances identified relative to expectations based on the National Functional Guidelines for Inorganic Data Review (EPA, February 1994, 48639). Data noncompliance is noted through the application of qualifiers (Attachment A) and reason codes (Attachment B) to the reported results. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as general indicators of data quality and should not be construed as a definitive identification of data usability.
- 1.3 Nothing in this SOP precludes the validator from going beyond the minimum requirements specified in this SOP. In order to address data quality issues in a data package, the validator may assign qualifiers using professional judgment. Implementation of this procedure may be followed by a more focused and data-use-specific evaluation of the data, especially if implementation of this SOP indicates that the data may contain technical deficiencies. The validator will note any need for a more focused validation on the Data-Validation Cover Sheet (Attachment C). The validator will use the Inorganic Data-Validation Checklist (Attachment D) to record the specific validation steps conducted.

2.0 SCOPE

- 2.1 All **RRES-R Personnel** shall implement this mandatory SOP when when evaluating routine inorganic analytical data.

- 2.2 Subcontractors performing work under the RRES-R Program's quality program shall follow this SOP.

3.0 TRAINING

- 3.1 **RRES-R Personnel** using this SOP shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.
- 3.2 **RRES-R Personnel** using this SOP shall document training in the RRES-R training database located at <http://erinternal.lanl.gov/Training/login.asp> in accordance with QP-2.2.
- 3.3 The responsible **supervisor** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.
- 3.4 All **data validators** implementing this SOP shall possess a minimum of a bachelor's degree in chemistry or one of the physical sciences and two years' experience in generating analytical data in an environmental analytical laboratory or two years' data-validation experience.
- 3.5 **Validators** who are not trained to this SOP shall work under the direct supervision of an experienced RRES-R Program validator. An experienced RRES-R Program validator shall review and sign the validator's work until ten data record packages for this data-validation SOP are satisfactorily validated.
- 3.6 **RRES-R Program validators** shall have demonstrated familiarity with the Environmental Protection Agency (EPA) national functional guidelines for data review.

4.0 DEFINITIONS

- 4.1 *Analyte*—Element, nuclide, or ion that a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.
- 4.2 *Continuing calibration verification (CCV)*—Check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A CCV is performed every 12 hours of operation or (for inorganics and high explosives [HEs]) every 10 injections (samples and/or quality control [QC] samples), whichever is more frequent, thus verifying the satisfactory performance of an instrument on a day-to-day basis. The continuing 12-hour calibration period assumes that the instrument has not been shut down since the initial calibration.
- 4.3 *Data validator*—Person who has met the minimum standards of training established by the RRES-R Program for data validation and who performs

data validation on behalf of the RRES-R Program (hereinafter referred to as the “validator”).

- 4.4 *Detect (inorganic and organic)*—Sample result above the method detection limit (MDL) reported by the contract analytical laboratory. The contract laboratory reports the concentration of the analyte in the sample.
- 4.5 *Duplicate analysis*—Analysis performed on one of a pair of identically prepared subsamples taken from the same sample. The subsamples can be created in the field (field duplicate samples) or in the laboratory (laboratory duplicate samples).
- 4.6 *Duplicate measurement*—A second measurement performed on a prepared sample under identical conditions to evaluate the variance in the measurement.
- 4.7 *Form 1*—Organic analysis data sheet for each individual sample that includes the sample information needed to identify the sample and the analytical results for the sample. See the SOW for analytical services (RFP [request for proposal] No. 9-XS1-Q4257) for a more complete definition.
- 4.8 *Holding time*—Maximum length of time that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions, and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.
- 4.9 *Inductively coupled plasma/atomic emission spectroscopy (ICPAES)*—ICPAES measures characteristic emission spectra by optical spectrometry. Samples are nebulized, and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.
- 4.10 *Inductively coupled plasma/mass spectroscopy (ICPMS)*—ICPMS measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized, and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a multichannel electron multiplier.
- 4.11 *Initial calibration*—Process used to establish the relationship between instrument response and analyte concentration at several analyte-

concentration values to demonstrate that an instrument is capable of acceptable analytical performance.

- 4.12 *Interference-check sample (ICS)*—Sample used to verify the contract analytical laboratory's interelement and background correction factors for inductively coupled plasma emission spectroscopy (ICPES) analyses. The ICS shall be analyzed a minimum of twice in each 8-hour shift or at the beginning and end of each analysis run, whichever is more frequent.
- 4.13 *Laboratory control sample (LCS)*—Known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.
- 4.14 *Laboratory duplicate sample*—Portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision during analysis. Each duplicate sample is equally representative of the original material. Duplicate analyses also are performed to generate data and to determine the long-term precision of an analytical method on various matrices.
- 4.15 *Laboratory qualifier (or laboratory flag)*—Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines (EPA 1994, 48639; EPA 1999, 66649).
- 4.16 *LANL data-validation qualifiers*—Data qualifiers defined by LANL and used in the RRES-R Program routine-validation process. Attachment A lists all the data qualifiers that are applicable to all analytical suites.
- 4.17 *LANL data-validation reason codes*—Codes applied to the sample data by data validators who are independent of the contract laboratory that performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier along with a description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate RRES-R Program SOP.
- 4.18 *Matrix spike (MS)*—An aliquot of sample spiked with a known concentration of target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. Spiking typically occurs before sample preparation and analysis.
- 4.19 *Method detection limit (MDL)*—Minimum concentration of a substance that can be measured and reported with known statistical confidence that the analyte concentration is greater than zero. The MDL is determined by analysis of samples of a given matrix type that contain the analyte after

the sample is subjected to the usual preparation and analyses. The MDL is used to establish detection status.

- 4.20 *Nondetect (inorganics)*—Sample result that is less than the MDL. The laboratory reports nondetects as undetected at the reporting limit (RL).
- 4.21 *Percent recovery (%R)*—Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.
- 4.22 *Preparation blank*—An analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. The preparation blank is used to assess the potential for contamination of samples during preparation and analysis.
- 4.23 *Relative percent difference (RPD)*—Measure used to assess the precision between parent sample results and their associated duplicate results. The RPD is calculated as follows:

$$|RPD| = \left| \frac{S - R}{\left(\frac{S + R}{2} \right)} \right| 100 ,$$

where

RPD = relative percent difference,

S = parent sample result, and

R = duplicate sample result.

The RRES-R Program criteria for the RPD is less than 20% for aqueous samples and less than 35% for soil samples when the sample concentrations are greater than or equal to five times the MDL. For samples with concentrations less than five times the MDL, but greater than the MDL, the control is +/-MDL. No precision criterion applies to samples with concentrations less than the MDL.

- 4.24 *Reporting limit (RL)*—Lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine analytical-laboratory operating conditions. The low point on a calibration curve should reflect this reporting limit. The RL is not used to establish detection status.
- 4.25 *Request number (RN)*—Identifying number assigned by the RRES-R Program to a group of samples that are submitted for analysis.

- 4.26 *Routine data*—Data generated using analytical methods that are identified as routine methods in the current RRES-R Program SOW for analytical services.
- 4.27 *Routine data validation*—Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is twofold: (1) to estimate the technical quality of the data relative to minimum national guidelines adopted by the RRES-R Program; (2) to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.
- 4.28 *Serial dilution sample*—1:5 dilution of a sample. The analyte concentration in the undiluted sample should be at least 50 times the MDL for a comparison to be made (100 times for ICPMS). If the analyte concentration is sufficiently high, the serial dilution sample should agree within 10% of the original sample. The serial dilution sample is used to measure physical or chemical interferences.
- 4.29 *Target analyte*—Element, chemical, or parameter of which the concentration, mass, or magnitude is quantified by a particular test.

5.0 RESPONSIBLE PERSONNEL

- Data Validator
- RRES-R Personnel
- Project Team Leader
- Supervisor
- User

6.0 PROCEDURE

The **validator** shall perform steps 6.1 through 6.12. Make any deviations from this SOP in accordance with QP-5.7 and/or SOP-01.01.

6.1 Verifying Data Package Completeness

1. Obtain the required current version of the Inorganic Data-Validation Checklist (Attachment A) from the RRES-R Program website ().
2. Obtain the data record packages that contain the sample data requiring validation from the Sample Management Office (SMO).
 - A. Prepare the Validation Cover Sheet (Attachment C) by completing the top part of the cover sheet and placing a check or

other mark adjacent to the analytical suites that are being validated.

- B. If any data are rejected, check the rejected box and notify the project chemist immediately.

Note: You may use a single cover sheet when validating multiple analytical suites under the same request number (RN).

Note: Use a separate sheet of paper if needed to document each deficiency identified beyond the scope of this procedure, including phone conversations with the analytical laboratory concerning these deficiencies. Attach these sheets to the Data - Validation Cover Sheet.

3. Verify that the following items are present in the data record package:

- A signed SMO, Chain of Custody (COC) record
- The case narrative
- The result forms (CLP Form 1 or equivalent) for each sample
- The QC forms (CLP forms 2A, 2B, 3, 4, 5A, 5B, 6, 7, 8 [GFAA only], 9 [ICPES only], 10, 11A, 11B, or equivalent) for water and/or soils, as appropriate
- The instrument readout (raw data) for the samples

4. IF the required documentation for the data record package is...	FOR...	THEN...
Complete,		<ul style="list-style-type: none">• Go to Step 6.
Missing,	< 6 mo.	<ul style="list-style-type: none">• Contact the analytical laboratory and/or the SMO.• Allow 3 business days for submittal.• Go to Step 5.
Missing,	= 6 mo.	<ul style="list-style-type: none">• Contact the analytical laboratory and/or the SMO.• Allow 10 business days for submittal.• Go to Step 5.

Note: To expedite the validation process, the validator should request that the laboratory forward the missing information via email or fax directly to the validator within 24 h of notification.

5.	IF the analytical laboratory...	THEN...
	Submits the documentation within the specified time period,	<ul style="list-style-type: none">• Go to Step 6.
	Does <u>not</u> submit documentation within the specified time period,	<ul style="list-style-type: none">• Notify the SMO for contract-compliance action.• Go to Step 6.

6. In the Data-Validation Cover Sheet Completeness Check section,
 - A. Record the presence or absence (“Yes” or “No”) of each item, as appropriate.
 - B. Indicate under the Comments/Problems section any samples whose data are missing from the data record package.
7. Photocopy the following items:
 - The form 1s from the analytical laboratory (used during the validation process).
 - The chain of custody forms.

Note: Do not record the data-validation qualifiers and the reason codes on the original form (Form 1).

Note: The validator must submit the photocopies of the items listed in Step 7 as attachments to the completed Inorganic Data-Validation Checklist. Each Form 1 must be initialed and dated by the validator; these initials and date must be present even if the validator accepts laboratory qualification.

6.2 Verifying Blank Results

Using forms provided by the analytical laboratory, verify the presence of the initial- and continuing-calibration blanks (ICB and CCB) and the preparation blank results.

Note: If additional validation forms are needed to record validation data for more than one blank, make additional copies of the appropriate forms.

1. IF the appropriate preparation blank, ICB, and CCB were ...	THEN...
Analyzed and reported for each sample matrix and/or analytical batch,	<ul style="list-style-type: none"> Record "No" on line 1 of the Inorganic Data-Validation Checklist. Go to Step 2.
<u>Not</u> included for each sample matrix and/or analytical batch,	<ul style="list-style-type: none"> Record "Yes" on line 1 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I4) on the individual sample Form 1. Go to Step 2.

2. IF the preparation blank was reported and ...	THEN...
<u>No</u> target analytes were detected in the preparation blank,	<ul style="list-style-type: none"> Record "No" on line 2 of the Inorganic Data-Validation Checklist. Go to Step 3.
Target analytes were detected in the preparation blank and either none of the target analytes detected in the preparation blank were detected in any associated samples <u>or</u> those analytes detected in both the preparation blank and the samples were detected in the samples at a level greater than five times the amount detected in the preparation blank,	<ul style="list-style-type: none"> Record "No" on line 2 of the Inorganic Data-Validation Checklist. Go to Step 3.

2. IF the preparation blank was reported and ...	THEN...
<u>Any</u> target analytes were detected in the preparation blank and the same target analytes were detected in the associated samples at a level less than or equal to five times the amount detected in the preparation blank,	<ul style="list-style-type: none"> Record "Yes" on line 2 of the Inorganic Data-Validation Checklist. Qualify affected results as not detected (U, I4a) on the individual sample on Form 1. Go to Step 6.3.

6.3 Verifying Calibrations

Verify the presence of the initial- and continuing-calibration verification (ICV and CCV) results using the forms supplied by the analytical laboratory.

1. IF ICV and CCV analysis documentation for each sample matrix and/or analytical batch is...	THEN...
Present,	<ul style="list-style-type: none"> Record "No" on line 4 of the Inorganic Data-Validation Checklist. Go to Step 2.
Missing,	<ul style="list-style-type: none"> Record "Yes" on line 4 of the Inorganic Data-Validation Checklist Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I16) on the individual sample Form 1. Go to Step 2.

**Table 6.3-1
Calibration Verification Limits**

Analytes	LAL	LWL	UWL	UAL
All (except Hg and CN)	75	90	110	125
Hg	65	80	120	135
CN	70	85	115	130
LAL—Lower Acceptable Limit, LWL—Lower Warning Limit, UWL—Upper Warning Limit, UAL—Upper Acceptable Limit, Hg—mercury, CN—cyanide				

2.	IF...	THEN...
	<u>All</u> the ICV or CCV percent recoveries (%Rs) are = the upper acceptably limit (UAL) listed in table 6.3-1 ,	<ul style="list-style-type: none"> Record “No” on line 5 of the Inorganic Data-Validation Checklist. Go to Step 3.
	<u>Any</u> ICV or CCV %R are > the UAL,	<ul style="list-style-type: none"> Record “Yes” on line 5 of the Inorganic Data-Validation Checklist and for the affected analytes. Qualify detected analytes as rejected (R, I16a) on the individual sample Form 1. Go to Step 3.
3.	IF...	THEN...
	<u>No</u> ICV %R is > the upper warning limit (UWL),	<ul style="list-style-type: none"> Record “No” on line 6 of the Inorganic Data-Validation Checklist. Go to Step 4.

3.	IF...	THEN...
	<u>Any</u> ICV or CCV %R is > UWL and = the UAL,	<ul style="list-style-type: none"> Record "Yes" on line 6 of the Inorganic Data-Validation Checklist. For the affected analytes qualify the result for each detected analyte as estimated with a potential positive bias (J+, I16b) in the associated samples on the individual sample Form 1. Go to Step 4.

4.	IF...	THEN...
	<u>No</u> ICV or CCV %R is < the lower warning limit (LWL),	<ul style="list-style-type: none"> Record "No" on line 7 of the Inorganic Data-Validation Checklist. Go to Step 5.
	<u>Any</u> ICV or CCV %R is < the LWL and = the lower acceptable limit (LAL),	<ul style="list-style-type: none"> Record "Yes" on line 7 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each detected analyte as estimated with a potential negative bias (J-, I16c) in the associated samples on the individual sample Form 1. For the affected analytes, qualify the results for each undetected analyte as estimated (UJ, I16c) in the associated samples on the individual sample Form 1. Go to Step 5.

5.	IF...	THEN...
	<u>No</u> ICV or CCV %R is < the LAL,	<ul style="list-style-type: none"> Record "No" on line 8 of the Inorganic Data-Validation Checklist. Go to Step 6.

5.	IF...	THEN...
	Any ICV or CCV %R is < the LAL,	<ul style="list-style-type: none"> Record "Yes" on line 8 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each analyte as rejected (R, I16d) in the associated samples. Go to Step 6.
6.	IF...	THEN...
	The calibration correlation coefficient is < 0.995 for multipoint calibration analytes (ICPMS, CN, wet chem.),	<ul style="list-style-type: none"> Record "Yes" on line 9 of the Validation Checklist. Qualify the results for all analytes as rejected (R, I16e) in the associated samples on the individual sample Form 1. Go to Section 6.4, "Verifying ICPEs Interference Check Sample Results."
	Multipoint calibrations are <u>not</u> used <u>or</u> if the calibration coefficient is acceptable,	<ul style="list-style-type: none"> Record "No" on line 9 of the Inorganic Data-Validation Checklist. Go to Section 6.4, "Verifying ICPEs Interference Check Sample Results."

6.4 Verifying ICPEs Interference Check Sample Results

Verify the presence of the ICS %R values using forms provided by the analytical laboratory. The ICS must contain the following analytes: Ag, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb (see note, below), V and Zn. The QC acceptance limits are $\pm 20\%$.

Note: If lead was analyzed by graphite furnace atomic absorption (GFAA), no ICS result is required. This information should be noted in the comment section of the Data-Validation Cover Sheet.

1.	IF the ICS documentation is...	THEN...
	<u>Present</u> for each sample matrix and/or analytical batch,	<ul style="list-style-type: none"> Record "No" on line 10 of the Inorganic Data-Validation Checklist. Go to Step 2.
	<u>Missing</u> for each sample matrix and/or analytical batch,	<ul style="list-style-type: none"> Record "Yes" on line 10 of the inorganic data-validation checklist. Contact the laboratory and the SMO to request the missing information (see Section 6.1 -4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I7) on the individual sample Form 1. Go to Step 2.

2.	IF...	THEN...
	<u>All</u> ICS %R is = 120%,	<ul style="list-style-type: none"> Record "No" on line 11 of the Inorganic Data-Validation Checklist. Go to Step 3.
	<u>Any</u> ICS analyte %R value is > 120%	<ul style="list-style-type: none"> Record "Yes" on line 11 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each detected analyte as estimated with a potential positive bias (J+, I7a) in the associated samples. Go to Step 3.

3.	IF...	THEN...
	<u>All</u> ICS %R is = 80%,	<ul style="list-style-type: none"> Record "No" on line 12 of the Inorganic Data-Validation Checklist. Go to Step 4.

3.	IF...	THEN...
	<u>Any</u> ICS %R is = 50% and < 80%,	<ul style="list-style-type: none"> Record "Yes" on line 12 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results for each detected analyte, as estimated with a potential negative bias (J-, I7b) in the associated samples on the individual sample Form 1, <p style="text-align: center;">OR</p> <p>For the affected analytes qualify the results for each undetected analyte as estimated (UJ, I7b) in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 4.

4.	IF...	THEN...
	<u>All</u> ICS %R values are = 50%,	<ul style="list-style-type: none"> Record "No" on line 13 of the Inorganic Data-Validation Checklist. Go to Section 6.5, "Verifying the Matrix Spike Results."
	<u>Any</u> ICS analyte %R value is < 50%,	<ul style="list-style-type: none"> Record "Yes" on line 13 of the Inorganic Data-Validation Checklist. For the affected analytes, qualify the results as rejected (R, I7c) for each analyte in the associated samples on the individual sample Form 1 Go to Section 6.5, "Verifying the Matrix Spike Results."

6.5 Verifying the Matrix Spike Results

Verify the presence of the matrix spike (MS) sample %R values using the forms provided by the analytical laboratory. The MS acceptance criteria are 75%–125%, inclusive for all spiked analytes.

Note: If the sample result is greater than four times the spike added for any analyte, these acceptance criteria do not apply (per the applicable methods) for that analyte.

1. IF an MS was...	THEN...
Analyzed on a sample associated with this request and the MS included all required analytes,	<ul style="list-style-type: none"> Record "No" on lines 14, 15, and 16 of the Inorganic Data-Validation Checklist. Go to Step 4.
<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 14 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I3) on the individual sample Form 1. Go to Step 2.

2. IF...	THEN...
If insufficient sample volume was submitted for analysis and no MS could be analyzed,	<ul style="list-style-type: none"> Record "Yes" on line 15 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I3a) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3a) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 3.
If the sample volume is sufficient,	<ul style="list-style-type: none"> Go to Step 3.

3.	IF the MS was performed on a...	THEN...
	Non-LANL sample,	<ul style="list-style-type: none"> Record "Yes" on line 16 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I3b) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3b) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 4.
	LANL sample,	<ul style="list-style-type: none"> Go to Step 4.

4.	IF...	THEN...
	All MS %R are = 150%,	<ul style="list-style-type: none"> Record "No" on line 17 of the Inorganic Data-Validation Checklist. Go to Step 5.
	Any MS %R is > 150%,	<ul style="list-style-type: none"> Record "Yes" on line 17 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated with a potential positive bias (J+, I3c) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3c) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 5.

5.	IF...	THEN...
	<u>All</u> MS %R are = 125%,	<ul style="list-style-type: none"> Record "No" on line 18 of the Inorganic Data-Validation Checklist. Go to Step 6.
	<u>Any</u> MS %R is > 125% and = 150%,	<ul style="list-style-type: none"> Record "Yes" on line 18 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated with a potential positive bias (J+, I3d) for each detected analyte in the associated samples on the individual sample Form 1. Go to Step 6.

6.	IF...	THEN...
	<u>All</u> MS %R = 75%,	<ul style="list-style-type: none"> Record "No" on line 19 of the Inorganic Data-Validation Checklist. Go to Section 6.6, "Verifying Duplicate Sample Analysis Results."
	<u>Any</u> MS %R is = 30% and is < 75%,	<ul style="list-style-type: none"> Record "Yes" on line 19 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated potential negative bias (J-, I3e) for each detected analyte in the associated samples on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>Qualify the affected results as estimated (UJ, I3e) for each undetected analyte in the associated samples on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 7.

7. IF...	THEN...
<u>All</u> MS %R are = 30%,	<ul style="list-style-type: none"> Record "No" on line 20 of the Inorganic Data-Validation Checklist. Go to Section 6.6, "Verifying Duplicate Sample Analysis Results."
<u>Any</u> MS %R is < 30%,	<ul style="list-style-type: none"> Record "Yes" on line 20 of the Inorganic Data-Validation Checklist. Qualify the affected results as rejected (R, I3f) for each analyte in the associated samples on the individual sample Form 1. Go to Section 6.6, "Verifying Duplicate Sample Analysis Results."

6.6 Verifying Duplicate-Sample Analysis Results

Verify the presence of the analytical laboratory duplicate-sample %R values using the forms provided by the analytical laboratory. If the sample and duplicate-sample results are greater than or equal to five times the RL, the duplicate-sample criterion for aqueous samples is an RPD less than or equal to 20%; the duplicate-sample criterion for solid samples is an RPD less than or equal to 35%. If either the sample or duplicate-sample value is less than five times the RL, a control limit must be used that is equal to the RL for water samples and two times the RL for solid samples.

1. IF a duplicate sample was...	THEN...
Analyzed on a sample associated with this request and the duplicate-sample analysis included all required analytes,	<ul style="list-style-type: none"> Record "No" on lines 21, 22, and 23 of the Inorganic Data-Validation Checklist. Go to Step 4.
<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 21 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information

	<p>(see Section 6.1 -4).</p> <ul style="list-style-type: none"> • If the laboratory cannot provide the missing information, qualify the affected results as estimated for duplicates and serial dilutions are not as critical to overall data usability. (J, I10/ UJ, I10) on the individual sample Form 1. • Go to Step 2.
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2.	IF...	THEN...
	An insufficient sample volume was submitted for analysis and no duplicate sample could be analyzed,	<ul style="list-style-type: none"> • Record "Yes" on line 22 of the Inorganic Data-Validation Checklist. • Qualify the affected results as estimated (J, I10a/UJ, I10a) on the individual sample Form 1. • Go to Step 3.

3.	IF the duplicate was performed on a...	THEN...
	Non-LANL sample,	<ul style="list-style-type: none"> • Record "Yes" on line 23 of the Inorganic Data-Validation Checklist. • Qualify the affected results as estimated (J, I10b/UJ, I10b) on the individual sample Form 1. • Go to Step 4.
	LANL sample,	<ul style="list-style-type: none"> • Go to Step 4.

4.	IF the...	THEN...
	Duplicate sample meets all QC criteria,	<ul style="list-style-type: none"> • Record "No" on lines 24 and 25. • Go to Section 6.7, "Verify Laboratory Control Sample Results."

Sample result and the duplicate sample result are each = five times the RL, <u>and</u> the RPD exceeds 20% for aqueous samples <u>or</u> 35% for soil samples,	<ul style="list-style-type: none"> Record "Yes" on line 24 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I10c/UJ, I10c) on the individual sample Form 1. Go to Step 5.
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5. If either the sample result or the duplicate sample result is less than 5 times the RL, and the difference between the sample result and the duplicate sample result is greater than the RL for water samples or greater than 2 times the RL for solid samples,
 - A. Record "Yes" on line 25 of the Inorganic Data-Validation Checklist.
 - B. Qualify the affected results as estimated (J, I10d/UJ, I10d) on the individual sample Form 1.
 - C. Go to Section 6.7, "Verifying Laboratory Control Sample Results."

6.7 Verifying Laboratory Control Sample Results

Verify the presence of the laboratory control sample (LCS) %R values using forms provided by the analytical laboratory. The LCS criteria to apply to soil and water samples are given in Table. 6.7-1.

Table 6.7-1
Laboratory Control Sample Recovery Criteria

Analyte	LAL	LWL	UWL
Soil	30	75	125
Water	50	80	120
All values in % recovery (%R)			

Note: The solid LCS recovery criteria do not apply to Ag or Sb.

1.	IF an appropriate LCS was...	THEN...
	Analyzed and reported,	<ul style="list-style-type: none"> Record "No" on line 26 of the Inorganic data-validation checklist. Go to Step 2.
	<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 26 of the inorganic data-validation checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as rejected (R, I6) on the individual sample Form 1. Go to Step 2.

2.	IF...	THEN...
	<u>All</u> LCS %R are = the LWL,	<ul style="list-style-type: none"> Record "No" on line 27 of the Inorganic Data-Validation Checklist. Go to Step 3.
	<u>Any</u> LCS %R values are > the UWL,	<ul style="list-style-type: none"> Record "Yes" on line 27 of the Inorganic Data-Validation Checklist. Qualify all detected results associated with the high LCS recovery in the affected samples as estimated with a potential high bias (J+, I6a) on the individual sample Form 1. Go to Step 3.

3.	IF...	THEN...
	<u>All</u> LCS %R are = the LWL,	<ul style="list-style-type: none"> Record "No" on line 28 of the Inorganic Data-Validation Checklist. Go to Step 4.
	<u>Any</u> LCS analyte %R value is = the LAL and < the LWL,	<ul style="list-style-type: none"> Record "Yes" on line 28 of the inorganic data-validation checklist. For each detected analyte qualify the affected results as estimated with a potential negative bias (J-, I6b) on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>For each undetected analyte qualify the affected results as estimated with a potential low bias (UJ, I6b) on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 4.

4.	IF...	THEN...
	<u>All</u> LCS %R are = the LWL,	<ul style="list-style-type: none"> Record "No" on line 29 of the Inorganic Data-Validation Checklist. Go to Section 6.8, "Verifying Serial Dilution Sample Results."
	<u>Any</u> LCS analyte %R value is < the LAL,	<ul style="list-style-type: none"> Record "Yes" on line 29 of the Inorganic Data-Validation Checklist. Qualify the affected results as rejected (R, I6c) on the individual sample Form 1. Go to Section 6.8, "Verifying Serial Dilution Sample Results."

6.8 Verifying Serial Dilution Sample Results

Verify the presence of the serial dilution sample %R values using forms provided by the analytical laboratory. A serial dilution must be analyzed for

inductively coupled plasma atomic emission spectroscopy (ICPAES) and for inductively coupled plasma mass spectroscopy (ICPMS). The serial dilution must be performed on a sample from each group of samples with a similar matrix type (for example, water, soil).

Note: Samples identified as field blanks cannot be used for the serial dilution sample.

Note: If the analyte concentration is sufficiently high (greater than 50 times the IDL for ICPAES and greater than 100 times the IDL for ICPMS), the serial dilution sample (a fivefold dilution) must then agree within a 10% relative difference of the original sample value after correction for the dilution.

Note: The qualifier for a missing serial dilution sample is J, estimated. This appears inconsistent with qualifying data with other missing QC documentation as R, rejected. The difference lies in the fact that the R qualifiers are assigned due to shortfalls in calibration requirements. The end user of the data should determine whether a greater impact is seen on sample results when a serial dilution sample is missing.

1. IF a serial dilution sample was...	THEN...
Analyzed and reported,	<ul style="list-style-type: none"> Record "No" on lines 30 and 31 of the Inorganic Data-Validation Checklist. Go to Step 3.
<u>Not</u> reported with this request,	<ul style="list-style-type: none"> Record "Yes" on line 30 of the Inorganic Data-Validation Checklist. Contact the laboratory and SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected results as estimated (J, I18/UJ, I18) on the individual sample Form 1. Go to Step 2.

2.	IF a serial dilution sample was performed on a...	THEN...
	Non-LANL sample,	<ul style="list-style-type: none"> Record "Yes" on line 31 of the Inorganic Data-Validation Checklist. Qualify the affected results as estimated (J, I18a/UJ, I18a) on the sample Form 1. Go to Step 3.
	LANL sample,	<ul style="list-style-type: none"> Go to Step 3.

3.	IF...	THEN...
	<u>All</u> serial dilution %R are = 10%,	<ul style="list-style-type: none"> Record "No" on line 32 of the Inorganic Data-Validation Checklist. Go to Section 6.9, "Verifying Holding Times."
	<u>Any</u> serial dilution sample %R values are > 10% <u>and</u> the analyte value is > 50 times the MDL in the original sample for ICPAES analytes (> 100 times the MDL for ICPMS),	<ul style="list-style-type: none"> Record "Yes" on line 32 of the Inorganic Data-Validation Checklist. Qualify the results as estimated (J, I18b/UJ, I18b) on the individual sample Form 1. Go to Section 6.9, "Verifying Holding Times."

6.9 Verifying Holding Times

Note: Holding times for metals (except cyanide and mercury in aqueous samples) are typically six months. The holding time for mercury in an aqueous sample is 28 days after the sample collection. The holding time for cyanide in an aqueous sample is 14 days after the sample collection. Applicable storage conditions are found in the current SOW for analytical services (LANL 1995b).

1.	IF...	THEN...
	<u>All</u> samples were analyzed within the prescribed holding time,	<ul style="list-style-type: none"> Record "No" on line 33 of the Inorganic Data-Validation Checklist. Go to Section 6.10, "Identifying the Detection Status."

<p><u>Any</u> of the samples were <u>not</u> analyzed within the prescribed holding time,</p>	<ul style="list-style-type: none"> Record "Yes" on line 33 of the Inorganic Data-Validation Checklist. For each detected analyte in the associated samples qualify the results as estimated with a potential negative bias (J-, I9) on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>For each undetected analyte in the associated samples, qualify the results as estimated (UJ, I9) on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Step 2.
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2. IF...	THEN...
<p><u>All</u> samples were analyzed fewer than two times the prescribed holding time,</p>	<ul style="list-style-type: none"> Record "No" on line 34 of the Inorganic Data-Validation Checklist. Go to Section 6.10, "Identifying the Detection Status."
<p><u>Any</u> sample was analyzed more than two times the prescribed holding time,</p>	<ul style="list-style-type: none"> Record "Yes" on line 34 of the Inorganic Data-Validation Checklist. For each detected analyte in the associated samples, qualify the results as estimated with a potential negative bias (J-, I9a) on the individual sample Form 1. <p style="text-align: center;">OR</p> <p>For each undetected analyte in the associated samples, qualify the results as rejected (R, I9a) on the individual sample Form 1.</p> <ul style="list-style-type: none"> Go to Section 6.10, "Identifying the Detection Status."

6.10 Identifying the Detection Status

Note: In order to meet the regulatory requirements imposed upon the RRES-R Program with the technology routinely available from the environmental laboratory community, the RRES-R Program requires analytical laboratories to report inorganic analytes as detected down to the MDL. In order to identify results below the RL and results with greater errors in quantitation, the laboratories have been instructed to apply a “B” flag to all results between the MDL and RL.

IF...	THEN...
<u>No</u> results are reported with a “B” flag,	<ul style="list-style-type: none">Record “No” on line 35 of the Inorganic Data-Validation Checklist.Go to Section 6.11, “Identifying Obvious Quality Deficiencies.”
<u>Any</u> results are reported by the contract laboratory with a “B” flag,	<ul style="list-style-type: none">Record “Yes” on line 35 of the inorganic data-validation checklist.For each detected analyte qualify affected results as estimated (J, I1) on the individual sample Form 1.Go to Section 6.11, “Identifying Obvious Quality Deficiencies.”

Note: A J,I1 qualifier is superseded by U,I4a.

6.11 Identifying Obvious Quality Deficiencies

IF...	THEN...
<u>Any</u> significant or obvious data-quality deficiencies during the data-validation process are noticed,	<ul style="list-style-type: none">Record “Yes” on line 36 of the Inorganic Data-Validation Checklist.Contact the analytical laboratory and SMO, if necessary to resolve the quality issue.Record the appropriate data qualifier based on the validator’s best professional judgment and apply reason code I19.Describe clearly the quality issue flagged on the Data-Validation Cover Sheet.

IF...	THEN...
	<ul style="list-style-type: none"> Go to Section 6.12, "Assembling the Data Record Package."
There are <u>no</u> obvious quality deficiencies outside of those covered by this SOP,	<ul style="list-style-type: none"> Record "No" on line 36 of the Inorganic Data-Validation Checklist. Go to Section 6.12, "Assembling the Data Record Package."

6.12 Assembling and Submitting the Data Record Package

1. Assemble the validation data record package to include the following items in the this order:
 - The completed, signed, and dated Data-Validation Cover Sheet.
 - The Inorganic Data-Validation Checklist completed in Sections 6.2 through 6.9.
 - Photocopies of the completed forms (Form 1) on which the validator recorded data-validation qualifier flags and reason codes.
 - Photocopies of the data record package chain of custody forms.
2. Attach the data-validation record package to the original data package and submit it to the SMO, in accordance with SOP-15.09.

7.0 LESSONS LEARNED

- 7.1 Before performing work described in this SOP, **RRES-R Personnel** should go to the Department of Energy Lessons Learned Information Services home page, located at <http://www.tis.eh.doe.gov/II/II.html>, and/or to the LANL Lessons Learned Resources web page, located at http://www.lanl.gov/projects/lessons_learned/, and search for applicable lessons.
- 7.2 During work performance and/or after the completion of work activities, **RRES-R Personnel**, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at http://www.lanl.gov/projects/lessons_learned/.

8.0 RECORDS

No records are submitted to the Records Processing Facility (RPF) when this procedure is completed. The items identified in Section 6.11 are a part of the

data record package submitted to the RPF from the SMO, in accordance with SOP-15.09, Chain of Custody for Analytical Data Packages.

9.0 REFERENCES

To properly implement this SOP, **RRES-R Personnel** should become familiar with the contents of the following documents located at http://erinternal.lanl.gov/home_links/Library_proc.shtml:

- EPA (U.S. Environmental Protection Agency), "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," Publication 9240.1-05-01, EPA-540/R-94/013, Office of Solid Waste and Emergency Response, Washington, DC, (February 1994).
- Los Alamos National Laboratory, "Environmental Restoration Project Statement of Work for Analytical Services," Revision 2, RFP Number 9-SX1-Q4257, Los Alamos National Laboratory, Los Alamos, New Mexico, (July 1995).
- QP-2.2, Personnel Orientation and Training
- QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities
- SOP-01.01, General Instructions for Field Investigations
- SOP-15.09, Chain of Custody for Analytical Data Packages

10.0 ATTACHMENTS

The **user** of this SOP may locate all forms associated with this procedure at <http://erinternal.lanl.gov/Quality/user/forms.asp>.

Attachment A: Laboratory Data-Validation Qualifier Flags, 1 page

Attachment B: Inorganic Data-Validation Reason Codes, 3 pages

Attachment C: Data-Validation Cover Sheet, 1 page

Attachment D: Inorganic Data-Validation Checklists, 1 page

Attachment E: List of Acronyms and Abbreviations, 1 page

Attachment A: Laboratory Data-Validation Qualifier Flags

- J The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual.
- J+ The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
- J- The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.
- U The analyte is classified “undetected.”
- UU The analyte is classified “undetected” with an expectation that the reported result is more uncertain than usual.
- R The analyte is classified “rejected” due to serious noncompliances regarding quality-control acceptance criteria.

Attachment B: Inorganic Data-Validation Reason Codes

Reason Code	Qualifier Detects	Qualifier non-Detects	Description
I1	J	N/A	Results for the affected analytes are considered estimated (J) because the results were between the estimated quantization limit and the method detection limit.
I3	R	R	Results of the affected analytes are considered rejected (R) because the MS was not analyzed with the samples for unspecified reasons.
I3a	J	UJ	Results of the affected analytes are considered estimated (J) because there was insufficient sample volume for an MS to be analyzed on a LANL sample.
I3b	J	UJ	Results of the affected analytes are considered estimated (J) because the MS was analyzed on a non-LANL sample.
I3c	J+	UJ	Results for the affected analytes are considered estimated and biased high (J+; UJ for undetected analytes) because the analyte was recovered above 150% in the associated spike sample.
I3d	J+	N/A	Results for the affected analytes are considered estimated and biased high (J+: detected analytes only) because the analyte was recovered above the upper acceptance level but less than 150% of the associated spike sample.
I3e	J-	UJ	Results for the affected analytes are considered estimated and biased low (J-; UJ for undetected analytes) because the analyte was recovered below the lower acceptance level but greater than 30% in the associated spike sample.
I3f	R	R	Results for the affected analytes are considered rejected (R) because the associated spike sample recovered less than 30%.
I4	R	R	Results for the affected analytes are considered rejected (R) because a preparation blank, ICB, or CCB was not analyzed with the samples for unspecified reasons.
I4a	U	N/A	Results for the affected analytes are regarded as undetected (U) because the results are less than five times the amount in the preparation blank.
I4b	U	N/A	Results for the affected analytes are regarded as undetected (U) because the results are less than five times the amount in the associated ICB or CCB.
I6	R	R	Results for the affected analytes are considered rejected (R) because the LCS was not analyzed with the samples for unspecified reasons.
I6a	J+	N/A	Results for the affected analytes are considered estimated and biased high (J+: detected analytes only) because the associated LCS was recovered above the upper warning limit.
I6b	J-	UJ	Results for the affected analytes are considered estimated and biased low (J-; UJ for undetected analytes) because the associated LCS was recovered below the lower warning limit but greater than or equal to the lower acceptable limit.
I6c	R	R	Results for the affected analytes are considered rejected (R) because the associated LCS recovered less than the lower acceptable limit.

Reason Code	Qualifier Detects	Qualifier non-Detects	Description
I7	R	R	Results for the affected analytes are considered rejected (R) because the ICS was not analyzed with the samples.
I7a	J+	N/A	Results for the affected analytes are considered estimated and biased high (J+: detected analytes only) because the associated ICS was recovered above the upper acceptable limit.
I7b	J-	UJ	Results for the affected analytes are considered estimated and biased low (J-; UJ for undetected analytes) because the associated ICS was recovered below the lower warning limit but greater than or equal to the lower acceptable limit.
I7c	R	R	Results for the affected analytes are considered rejected (R) because the associated ICS recovered less than the lower acceptable limit.
I9	J-	UJ	Results for the affected analytes are considered estimated with a potential low bias (J-; UJ for undetected analytes) because the samples were analyzed after the appropriate hold time had passed.
I9a	R	R	Results for the affected analytes are considered rejected (R) because the samples were analyzed - after a period had elapsed equal to or greater than twice the hold time.
I10	J-	R	Results for the affected analytes are considered estimated with a potential low bias/rejected (J-/R) because the duplicate sample was not analyzed with the samples for unspecified reasons.
I10a	J	UJ	Results for the affected analytes are considered estimated (J/UJ) because there was insufficient sample volume for a duplicate sample to be (taken and) analyzed on from a LANL sample.
I10b	J	UJ	Results for the affected analytes are considered estimated (J/UJ) because the duplicate sample was analyzed on a non-LANL sample.
I10c	J	UJ	Results for the affected analytes are considered estimated (J; UJ for undetected analytes) because both the sample and duplicate sample results were greater than or equal to five times the RL and the duplicate RPD was greater than 20% for water samples and greater than 35% for soil samples.
I10d	J	UJ	Results for the affected analytes are considered estimated (J; UJ for undetected analytes) because either the sample or duplicate sample results or both were greater than or equal to 5 times the RL, and the difference between the samples is greater than the RL for water samples or greater than 2 times the RL for soil samples.
I16	R	R	Results of the affected analytes are considered rejected (R) because an ICV or CCV was not analyzed with the samples.
I16a	R	R	Results for the affected analytes are considered rejected (R) because the associated ICV or CCV was recovered above the upper acceptable limit.
I16b	J+	N/A	Results for the affected analytes are considered estimated and biased high (J+: detected analytes only) because the associated ICV or CCV was recovered above the upper warning limit but is less than or equal to the upper acceptable limit.

Reason Code	Qualifier Detects	Qualifier non-Detects	Description
I16c	J-	UJ	Results for the affected analytes are considered estimated and biased low (J-; UJ for undetected analytes) because the associated ICV or CCV was recovered below the lower warning limit but is greater than the lower acceptable limit.
I16d	R	R	Results for the affected analytes are considered rejected (R) because the associated ICV or CCV was recovered but is below the lower acceptable limit.
I16e	R	R	Results for the affected analytes are considered rejected (R) because the associated multipoint calibration correlation coefficient is less than 0.995.
I18	J	UJ	Results for the affected analytes are considered estimated (J; UJ for undetected analytes) because a serial dilution sample was not analyzed with the samples.
18a	J	UJ	Results of the affected analytes are considered estimated (J; UJ for undetected analytes) because the serial dilution sample was analyzed on a non-LANL sample.
I18b	J	UJ	Results of the affected analytes are considered estimated (J; UJ for undetected analytes) because the serial dilution sample RPD was greater than 10% and the sample result was greater than 50 times the MDL (>100 times the MDL for ICPMS).
I19	See comments	See comments	<div> The validator identified quality deficiencies in the reported data that require qualification. Please see the Data-Validation Cover Sheet for specific details. </div> <div> Apply the appropriate qualifier to identify the effect of the quality deficiency on the reported data. </div>

Attachment C: Data-Validation Cover Sheet



Rejected Data

Section I

Request Number: _____ Validation Date: _____ Rejected Data: _____

Contract Laboratory Name: _____

Validator: _____ Organization: _____

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ High Explosives
☐ Semivolatile Organics ☐ Inorganics
☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☐ Radiochemistry

Other (describe): _____

Section II—Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. Chain-of-custody form(s)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Raw/BSS data
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. Case narrative	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. Quality control forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. Sample result forms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. Quantitation reports
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. Sample chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. TICs forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. Standard chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. TICs mass spectra

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: _____ Date: _____

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Attachment D: Inorganic Data-Validation Checklist

Yes	No	(check one)	Assign qualifier listed below if criteria = Yes	
			Detected analyte	Undetected analyte
<input type="checkbox"/>	<input type="checkbox"/>	1. The preparation Blank (PB), ICB, or CCB was not analyzed with the samples.	R, I4	R, I4
<input type="checkbox"/>	<input type="checkbox"/>	2. The analyte detected in PB and the sample result for the analyte =5x the amount in PB.	U, I4a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	3. The initial or continuing calibration verification (ICV or CCV) was not analyzed with the samples.	R, I16	R, I16
<input type="checkbox"/>	<input type="checkbox"/>	4. The ICV or CCV recovery is >UAL.	R, I16a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	5. The ICV or CCV recovery is >UWL and =UAL.	J+, I16b	N/A
<input type="checkbox"/>	<input type="checkbox"/>	6. The ICV or CCV recovery is <LWL but =LAL.	J-, I16c	UJ, I16c
<input type="checkbox"/>	<input type="checkbox"/>	7. The ICV or CCV recovery is <LAL.	R, I16d	R, I16d
<input type="checkbox"/>	<input type="checkbox"/>	8. For ICPMS and CN analyses: The correlation coefficient is <0.995?	R, I16e	R, I16e
<input type="checkbox"/>	<input type="checkbox"/>	9. The interference check sample (ICS) was not analyzed with the samples.	R, I7	R, I7
<input type="checkbox"/>	<input type="checkbox"/>	10. The ICS recovery is >120%.	J+, I7a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	11. The ICS recovery is =50% and <80%.	J-, I7b	UJ, I7b
<input type="checkbox"/>	<input type="checkbox"/>	12. The ICS recovery is <50%.	R, I7c	R, I7d
<input type="checkbox"/>	<input type="checkbox"/>	13. The MS was not analyzed with the sample without explanation.	R, I3	R, I3
<input type="checkbox"/>	<input type="checkbox"/>	14. Insufficient sample volume for MS analyses was provided.	J, I3a	UJ, I3a
<input type="checkbox"/>	<input type="checkbox"/>	15. An MS analyses was performed on a non-LANL sample.	J, I3b	UJ, I3b
<input type="checkbox"/>	<input type="checkbox"/>	16. The MS recovery was > 150%*.	J+, I3c	UJ, I3c
<input type="checkbox"/>	<input type="checkbox"/>	17. The MS recovery was >125% and =150%.	J+, I3d	N/A
<input type="checkbox"/>	<input type="checkbox"/>	18. The MS recovery was =30% and <75%.	J-, I3e	UJ, I3e
<input type="checkbox"/>	<input type="checkbox"/>	19. The MS recovery was <30%.	R, I3f	R, I3f
<input type="checkbox"/>	<input type="checkbox"/>	20. A duplicate sample was not analyzed without explanation.	J, I10	UJ, I10
<input type="checkbox"/>	<input type="checkbox"/>	21. Insufficient sample volume for duplicate sample analysis was provided.	J, I10a	UJ, I10a
<input type="checkbox"/>	<input type="checkbox"/>	22. A duplicate sample was performed on a non-LANL sample.	J, I10b	UJ, I10b
<input type="checkbox"/>	<input type="checkbox"/>	23. Both the sample and the duplicate are =5x RL, and the RPD is >20 for water samples or >35 for soil samples	J, I10c	UJ, I10c
<input type="checkbox"/>	<input type="checkbox"/>	24. Either the sample or Dup is =5x the RL and the sample and Dup results are not within ±1x the RL for water or ±2x the RL for soil.	J, I10d	UJ, I10d
<input type="checkbox"/>	<input type="checkbox"/>	25. The LCS was not analyzed with the samples.	R, I6	R, I6
<input type="checkbox"/>	<input type="checkbox"/>	26. The LCS recovery was >the UWL.	J+, I6a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	27. The LCS recovery was =LAL and <LWL.	J-, I6b	UJ, I6b
<input type="checkbox"/>	<input type="checkbox"/>	28. The LCS recovery was <the LAL.	R, I6c	R, I6c
<input type="checkbox"/>	<input type="checkbox"/>	29. The serial dilution sample was not analyzed with the samples.	J, I18	UJ, I18
<input type="checkbox"/>	<input type="checkbox"/>	30. The serial dilution sample was performed on a non-LANL sample.	J, I18a	UJ, I18a
<input type="checkbox"/>	<input type="checkbox"/>	31. The RPD between the sample and the serial dilution sample is >10, and the undiluted sample result is >50x the RL(>100x for ICPMS).	J, I18b	UJ, I18b
<input type="checkbox"/>	<input type="checkbox"/>	32. The sample was analyzed past the appropriate hold time.	J-, I9	UJ, I9
<input type="checkbox"/>	<input type="checkbox"/>	33. The sample was analyzed past double the hold time.	J-, I9a	R, I9a
<input type="checkbox"/>	<input type="checkbox"/>	34. B-flagged sample results are present.	J, I1	N/A
<input type="checkbox"/>	<input type="checkbox"/>	35. Other obvious data quality issues are identified.	__, I19	__, I19

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* If the sample result is >4x the spike added, MS recovery criteria do not apply.

Attachment E: List of Acronyms and Abbreviations

CCB	continuing calibration blank	IDL	instrument detection limit
CCV	continuing calibration verification	LAL	lower acceptable limit
CLP	Contract Laboratory Program	LANL	Los Alamos National Laboratory
CN	cyanide	LCS	laboratory control sample
COC	chain of custody	LWL	lower warning limit
EPA	Environmental Protection Agency	MDL	method detection limit
ER	environmental restoration	MS	matrix spike
HE	high explosive	N/A	not applicable
Hg	mercury	QC	quality control
FSF	field support facility	QP	quality procedure
GFAA	graphite furnace atomic absorption	%R	percent recovery
ICB	initial calibration blank	PB	preparation blank
ICPAES	inductively coupled plasma atomic emission spectroscopy	RFP	request for proposal
ICPES	inductively coupled plasma emission spectroscopy	RPD	relative percent difference
ICPMS	inductively coupled plasma mass spectroscopy	RL	reporting limit
ICS	interference check sample	RN	request number
ICV	initial calibration verification	SMO	Sample Management Office
		SOP	standard operating procedure
		SOW	statement of work
		UAL	upper acceptable limit
		UWL	upper warning limit